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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001] This invention relates to the manufacture approach of an N and N'- diacyl-N'-t-alkyl hydrazine. In a detail, this invention is set more at the 1st process. A ketone or an aldehyde Make it react with N'-t-alkyl hydrazines or those suitable acid addition salts (acid addition salt), and an N-alkylidene-N'-t-alkyl hydrazone is formed. Subsequently, in the 2nd process, acylate by the aromatic series acid chloride, and an N-alkylidene-N'- acyl-N'-t-alkyl hydrazone is formed. In the 3rd process, hydrolyze from a mild acid (mild acid), and an N'- acyl-N'-t-alkyl hydrazine is formed. It is related with the manufacture approach of the aforementioned diacyl hydrazine which acylates by the 2nd aromatic series acid chloride in the 4th process, and forms an N and N'- diacyl-N'-t-alkyl hydrazine. It is known that such a compound has the insect-killing property which was excellent to the insect of Lepidoptera and the Coleoptera. This invention relates to an N [of the specific class manufactured by the approach of this invention], and N'- diacyl-N'-t-alkyl hydrazine further.

[0002] Desired intermediate field and a desired insect-killing product were given with good selectivity, and in order that retrieval of the economically excellent approach might obtain the intermediate field and the product of a high grade by high yield, might decrease the cost which accompanies manufacture and it and might decrease the amount of the raw material used for desired insect-killing product generation, and the accompanying by-product, it was continued. The well-known approach for manufacture of an N and N'- diacyl-N'-t-alkyl hydrazine is indicated by U.S. Pat. No. 5,117,057. However, a special reagent like the problem of the selectivity of a desired intermediate product or dialkyl, diaryl one, or JIARU alkyl dicarbonate needed to be used for the approach currently indicated by it. European Patent 0232075B makes a ketone or an aldehyde react with N'-t-alkyl hydrazines or those suitable acid addition salts at the 1st process. Form an N-alkylidene-N'-t-alkyl hydrazone and, subsequently it acylates by the aromatic series acid chloride at the 2nd process. An N-alkylidene-N'- acyl-N'-t-alkyl hydrazone is formed. It hydrolyzes at the 3rd process with a dilute sulfuric acid or strong acid like dilute hydrochloric acid, an N'- acyl-N'-t-alkyl hydrazone is formed, it acylates by the 2nd aromatic series acid chloride at the 4th process, and the approach of forming an N and N'- diacyl-N'-t-alkyl hydrazine is indicated. However, use of the strong acid in the 3rd process of European Patent 0232075B causes formation of N-acylhydrazine by the irreversible loss of the side reaction which is not desirable, i.e., t-alkyl part. [0003] The purpose of this invention is offering the manufacture approach of an N and N'diacyl-N'-t-alkyl hydrazine which does not produce the above-mentioned problem. In the 3rd process of this invention approach which hydrolyzes an N-alkylidene-N'-acyl-N'-t-alkyl hydrazone alternatively and is made into an N-acyl-N'-t-alkyl hydrazine If a mild acid like a maleic acid, an OKISA rucksack acid (oxalic acid), phosphoric acid, an acetic acid, and Para toluenesulfonic acid is used It found out that this compound could be obtained by high yield, without generating the N'-acylhydrazine which is the by-product which is not desirable. Although not restrained by the theory, it is thought that the balance of the hydrolysis use of a mild acid is indicated to be by the following formulas (the inside of a formula, and R1, R2, R3 and B are the below-mentioned connoisseurs) is advanced. The conclusion of a reaction will be promoted if distillation removes the ketone or aldehyde to generate.

[Formula 30]

$$C=N-N-C-B \qquad \begin{array}{c|c} R^1 & & \\ \hline & & \\$$

[0005] Use of strong acid, such as a hydrochloric acid and a sulfuric acid, promotes irreversible decomposition of the intermediate field of the request by the loss of t-alkyl group. As shown in the following formulas, there is possibility to a hydrazine and a carboxylic acid of decomposing, further succeedingly.

[0006]

[Formula 31]

$$R^2$$
 $C=N-N-C-B$ 強敵 H $N-N-C-B$ R^2 R^3 $B-COOH + H_2NNH_2$ H $N-N-C-B$ H $N-N-C-B$ H $N-N-C-B$

[0007] This invention is a structure expression [0008]. [Formula 32]

[0009] R1 among [type the 3rd class (C4-C8) alkyl group, and A and B; halo which is independently chosen from phenyl, naphthyl, or the group that consists of the following radicals, respectively and which is the same, the phenyl permuted by different 1 thru/or three different substituents, or naphthyl, Cyano ** nitroglycerine, hydroxy ** mercapto, thiocyanate, alkyl (C1-C<SUB> 4), Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Amino, alkylamino (C1-C4), the JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently, Alkyl carbamoyl, the JI (C1-C4) alkyl carbamoyl in which each alkyl group has one to four carbon atoms independently, (C1-C4) Cyano (C1-C4) alkyl, alkoxy (C1-C4) (C1-C4) alkyl, The alkenyl, alkadienyl (C4-C6), alkynyl (C2-C6), (C2-C6) Alkyl dithio NETO, alkyl (C1-C4) carbonyl thio, (C1-C4) The Tori (C1-C4) alkyl silyl to which each alkyl group has one to four carbon atoms independently in a list, Phenyl and a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) In the phenyl which is chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which was permuted by the same or different 1 thru/or two substituents, phenoxy, and a list, a halo, Cyano ** nitroglycerine, hydroxy ** (C1-C4)

alkyl, alkoxy (C1-C4) ** Halo (C1-C2) alkyl, halo (C1-C2) alkoxy ** (C1-C4) alkylthio, Alkyl sulfinyl, an alkyl (C1-C4) sulfonyl, (C1-C4) Carboxy, the formyl, alkyl (C1-C4) carbonyl, alkoxy (C1-C4) carbonyl, Alkanoloxy, amino, alkylamino (C1-C4), (C1-C4) And the phenoxy which is chosen from the group which each alkyl group becomes from the JI (C1-C4) alkylamino which has one to four carbon atoms independently and which was permuted by the same or different 1 thru/or two substituents, In benzyl and a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Benzyl, phenoxy carbonyl which are chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which were permuted by the same or different 1 thru/or two substituents, In a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Phenoxy carbonyl, phenylthio which are chosen from the group which consists of amino, alkylamino (C1-C4), and II (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which were permuted by the same or different 1 thru/or two substituents, In a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) The phenylthio which is chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which was permuted by the same or different 1 thru/or two substituents, In phenyl (C1-C4) alkyl and a list, a halo, cyano ** nitroglycerine, hydroxy ** Alkyl, alkoxy (C1-C4) ** halo (C1-C2) alkyl, (C1-C4) Halo (C1-C2) alkoxy ** (C1-C4) alkylthio, alkyl (C1-C4) sulfinyl, An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Phenyl which is chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylaming in which each alkyl group has one to four carbon atoms independently and by which the phenyl ring was permuted by the same or different 1 thru/or two substituents (C1-C4) Alkyl; when two adjoining locations of a phenyl ring are permuted by the alkoxy group] which these radicals become together and can form the dioxo llano (methylene dioxy) or JIOKISANO (1, 2-ethylene dioxy) heterocycle of 5 or 6 members, An N [which is come out of and expressed], and N'- diacyl-N'-t-alkyl hydrazine compound, Or it is the manufacture approach of the salt permitted agriculturally, and is i. The acid addition salt to which a formula, the 3rd class (C4-C8) alkyl hydrazine expressed with H2NNHR1, or this the 3rd class (C4-C8) alkyl hydrazine is equivalent, and formula [0010] [Formula 33]

[0011] The ketone or aldehyde which **** is made to react under existence of a base, and it is a formula and [0012].

[0013] The intermediate product which has [R1 is as having given the definition previously among a formula, R2 is a hydrogen atom or (C1-C3) alkyl, and R3 is alkyl (C1-C6)] is formed, and it is ii. It is said intermediate product in Process i A formula and [0014] [Formula 35]

[0015] It is made to react under existence of the acid chloride which ****, and a base, and is a formula and [0016].

[Formula 36]

[0017] The intermediate product which has [the inside of a formula, and R1, R2, R3 and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with a mild acid, and it is a formula and [0018].

[0019] The intermediate product which has [the inside of a formula, and R1 and B are as having given the definition previously] is formed, and it is iv. It is said intermediate product A formula and [0020] [Formula 38]

[0021] It is made to react under existence of the 2nd acid chloride and base which ****, and is a formula and [0022].

[0023] The manufacture approach of an N and N'- diacyl-N'-t-alkyl hydrazine compound which forms an N [which is expressed with [the inside of a formula, and R1, A and B are as having given the definition previously] and N'- diacyl-N'-t-alkyl hydrazine compound is offered. [0024] The definition of the vocabulary used by this invention is as follows. A halo says chloro, fluoro, BUROMO, or iodine. (C1-C4) Alkyl says a straight chain or a branched chain alkyl group, for example, says methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, sec-butyl, or t-butyl. The 3rd class (C4-C8) alkyl says t-butyl [for example,], 1, and 1-dimethyl pentyl and 1, 1, 3 and 3, and tetramethylbutyl. (C2-C6) The alkenyl says vinyl, aryl, or 2-butene-1-IRU. (C4-C6) Alkadienyl says 2 and 4-pentadiene-1-IRU. (C2-C6) Alkynyl says propargyl. (C1-C4) Alkoxy **, for example, methoxy, and ethoxy ** or isopropoxy is said. (C1-C4) Alkoxy (C1-C4) alkyl says methoxymethyl or 2-methoxy ethyl. Halo (C1-C2) alkyl says trifluoromethyl, 1, 1, 2 and 2, 2-pentafluoroethyl, or chloromethyl. Halo (C1-C2) alkoxy **, for example, difluoro methoxy, is said. (C1-C4) Alkylthio says a methylthio. (C1-C4) Alkyl carbonyl says for example, methyl carbonyl (acetyl). (C1-C4) Alkoxy carbonyl says ethoxycarbonyl. (C1-C4) Alkanoloxy says for example, methyl carbonyloxy (acetoxy). (C1-C4) Alkylamino says for example, isobutyl amino. JI (C1-C4) alkylamino says dimethylamino or N-methyl-N-ethylamino. (C1-C4) Alkyl carbamoyl says for example, n-butylcarbamoyl. JI (C1-C4) alkyl carbamoyl says for example, dimethyl carbamoyl or N-methyl-N-ethyl carbamoyl. (C1-C4) Alkyl sulfinyl says for example, methyl sulfinyl. (C1-C4) An alkyl sulfonyl says methylsulfonyl. (C1-C4) Alkyl dithio NETO says for example, methyl dithio NETO. (C1-C4) Alkyl carbonyl thio says for example, ethyl carbonyl thio. Tori (C1-C4) alkyl silyl says trimethylsilyl or diethyl methyl silyl. Phenyl

(C1-C4) alkyl says benzyl or 4-chloro phenethyl. Methylene dioxy says a -OCH2O-part. 1 and

2-ethylene dioxy says a -O-CH2CH2O-part.

[0025] As an example of the typical acid chloride (ACOCl and BCOCl) which can be used in this invention approach A benzoyl chloride, 3-methyl benzoyl chloride, 3, 5-dimethyl benzoyl chloride, 3, 5-dichloro benzoyl chloride, 4-ethyl benzoyl chloride, 4-chloro benzoyl chloride, A 2-methyl-3-methoxy benzoyl chloride, 3, 5-dichloro benzoyl chloride, 3-chloro benzoyl chloride, 4-cyano benzoyl chloride, 3-nitro benzoyl chloride, 2, 3-dimethyl benzoyl chloride, 3-methoxy benzoyl chloride, 3-fluoro benzoyl chloride, 4-(t-butyl) benzoyl chloride, 2, 4-dichloro benzoyl chloride, 3-(trifluoromethyl) benzoyl chloride, 2-acetoxy benzoyl chloride, 4-(4-chloro phenethyl) benzoyl chloride, 1-chlorination naphthoyl, 4-isopropyl benzoyl chloride, 2, a 6-difluoro benzoyl chloride, 2-(difluoro methoxy) benzoyl chloride, 4-acetyl benzoyl chloride, 3-(dimethylamino) benzoyl chloride, a 2-nitro-4-(4-trifluoromethyl) (-2-chloro phenoxy) benzoyl chloride, A phenylthio benzoyl chloride, 4-(2-fluoro benzyl) benzoyl chloride, There are 4-(4-chloro benzoyl) benzoyl chloride, 4-(phenoxy carbonyl) benzoyl chloride, the 2-methyl -3, 4-(1, 2-ethylene dioxy) benzoyl chloride, the 2-methyl -3, a 4-(methylene dioxy) benzoyl chloride, etc.

[0026] As an example of the typical 3rd class alkyl hydrazine (H2NNHR1) which can be used in this invention approach, there is an acid addition salt like those hydrogen chloride salts, such as t-butyl hydrazine, 1, and 1-dimethyl pentyl hydrazine, 1, 1 and 3, and 3-tetramethyl BUCHIRUHIDORAJIN,

for example, t-butyl hydrazine hydrogen chloride salt etc.

[0027] As an example of a typical N [which is manufactured by this invention approach], and N'diacyl-N'-t-alkyl hydrazine (ACONHN(R1) COB) N'-t-butyl-N, an N'-dibenzoyl hydrazine, an N'-t-butyl-N-(4-chloro benzoyl)-N'-benzoyl hydrazine, An N'-t-butyl-N-(4-ethyl benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(2-methyl-3-methoxy benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(4-ethyl benzoyl)-N'-(3-methylbenzoyl) hydrazine, An

N'-t-butyl-N-(2-ethyl-3-methoxy benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(2,

3-dimethylbenzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(2,

3-dimethylbenzoyl)-N'-(3-methylbenzoyl) hydrazine, An N'-t-butyl-N-(4-ethyl benzoyl)-N'-(3, 5-dichlorobenzoyl) hydrazine, An N'-t-butyl-N-(2-methyl-3, 4-(1, 2-ethylene dioxy) benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(2-methyl -3, 4-(methylene dioxy) benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(2-fluoro benzoyl)-N'-benzoyl hydrazine, An N'-t-butyl-N-(1-naphthoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-(1 and 1-dimethyl

pentyl)-N-(4-chloro benzoyl)-N'-benzoyl hydrazine, And there is an

N'-(1,1,3,3-tetrametylbutyl)-N-(2-methyl-3-methoxy benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine etc. [0028] As an example of the typical ketone (R2COR3) which can be used in the process i of this invention approach, there are an acetone, a methyl ethyl ketone, a diethyl ketone, a methyl n-propyl ketone, methyl isopropyl ketone, an ethyl n-propyl ketone, an ethyl isopropyl ketone, a JI n-propyl ketone, diisopropyl keton, a methyl n-butyl ketone, methyl isobutyl ketone, a methyl t-butyl ketone, an ethyl sec-butyl ketone, an ethyl isoamyl ketone, a methyl n-hexyl ketone, etc. The sum total carbon number of a desirable ketone is six or less ketone. The sum total carbon number of a more desirable ketone is four or less ketone. The most desirable ketone is an acetone.

[0029] As an example of the typical aldehyde (R3CHO) which can be used in the process i of this invention approach, there are an acetaldehyde, propionaldehyde, a butyraldehyde, isobutyraldehyde,

valeric aldehyde (n-valeraldehyde), etc.

[0030] As an example of the typical mild acid which can be used in hydrolysis of the process iii of this invention approach, there are a maleic acid, an OKISA rucksack acid, phosphoric acid, an acetic acid, Para toluenesulfonic acid, formic acid, a chloroacetic acid, an alt.-nitro benzoic acid, a tartaric acid, citric

[0031] As an example of the base which can be used in the processes i, ii, and iv of this invention approach, tertiary amine like triethylamine, a pyridine, potassium carbonate, a sodium carbonate, a sodium hydrogencarbonate, a potassium hydrogencarbonate, a sodium hydroxide, potassium

hydroxides, and such mixture exist.

[0032] As an example of the solvent which can be used useful in the processes i, ii, and iv of this invention approach, a methanol, ethanol, n-propyl alcohol, alcohol like isopropyl alcohol, ester like n-butyl acetate, a methylene chloride, toluene, a tetrahydrofuran, an acetonitrile, water, the ether, and those mixture exist.

[0033] As an example of the solvent which can be used useful in the process iii of this invention approach, a methanol, ethanol, n-propyl alcohol, alcohol like isopropyl alcohol, toluene, a tetrahydrofuran, an acetonitrile, water, the ether like dioxane, and those mixture exist. [0034] The approach of this invention is [about]. -It is 20 to about 100 degrees C in temperature, and can carry out with atmospheric pressure mostly. If it is a request, it can also carry out under pressurization or reduced pressure. The range of the temperature used in this invention approach is about 0 to about 90 degrees C more preferably.

[0035] Although the reactant of an equimolecular amount is used substantially, if it is a request, a small amount of [abundant or] reactant can also be used. Generally, per 1Eq of aromatic series acid chlorides of starting material and about 1.0 to about 1.5Eq base are used. When using the acid addition salt of the 3rd class alkyl hydrazine, additional use of the 1 moreEq base is carried out.

[0036] Although modification of this invention approach may be needed with the concrete reactant functional group of A and/or B substituent, such modification is obvious for this contractor. The approach of manufacturing a permissible salt agriculturally included by the manufacture manufactured by the approach of this invention is well-known to this contractor. It sets in the desirable mode of this invention, and is a structure expression [0037].

[0038] R1 is t-butyl, 1, and 1-dimethyl pentyl or 1,1,3,3-tetrametylbutyl among [type. A and B; halo which is independently chosen from phenyl, naphthyl, or the group that consists of the following radicals, respectively and which is the same, the phenyl permuted by different 1 thru/or three different substituents, or a naphthyl group, Cyano ** nitroglycerine, thiocyanate, alkyl (C1-C4), alkoxy (C1-C4) ** Halo (C1-C2) alkyl, halo (C1-C2) alkoxy ** (C1-C4) alkylthio, Alkyl sulfinyl, an alkyl (C1-C4) sulfonyl, (C1-C4) Carboxy, the formyl, alkyl (C1-C4) carbonyl, alkoxy (C1-C4) carbonyl, Alkanoloxy, the JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently, (C1-C4) Carbamoyl, alkyl (C1-C4) carbamoyl, the JI (C1-C4) alkyl carbamoyl in which each alkyl group has one to four carbon atoms independently, Cyano (C1-C4) alkyl, alkoxy (C1-C4) (C1-C4) alkyl, The alkenyl, alkadienyl (C4-C6), alkynyl (C2-C6), (C2-C6) Alkyl dithio NETO, alkyl (C1-C4) carbonyl thio, (C1-C4) The Tori (C1-C4) alkyl silyl in which each alkyl group has one to four carbon atoms independently, Phenyl, phenoxy, bay ZOIRU, phenyl (C1-C2) alkyl, In a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Each alkyl group independently and one to four carbon atoms Phenyl (C1-C2) alkyl which is chosen from the group which consists of JI (C1-C4) alkylamino which it has and by which the phenyl ring was permuted by the same or different 1 thru/or two substituents; when two adjoining locations of a phenyl ring are permuted by the alkoxy group] which these radicals become together and can form the dioxo llano or JIOKISANO heterocycle of 5 or 6 members, An N [which is come out of and expressed], and N'diacyl-N'-t-alkyl hydrazine compound, Or it is the manufacture approach of the salt permitted agriculturally, and is i. The acid addition salt to which a formula, the 3rd class (C4-C8) alkyl hydrazine expressed with H2NNHR1, or this the 3rd class (C4-C8) alkyl hydrazine is equivalent, and formula [0039]

[Formula 41]

[0040] The ketone which **** is made to react under existence of a base, and it is a formula and [0041]. [Formula 42]

$$C = N - N - H$$

[0042] The intermediate product which has [R1 is as having given the definition previously among a formula, R2 is alkyl (C1-C3), and R3 is alkyl (C1-C4)] is formed, and it is ii. It is said intermediate product in Process i A formula and [0043]

[Formula 43]

[0044] It is made to react under existence of the acid chloride which ****, and a base, and is a formula and [0045].

[Formula 44]

$$\begin{array}{c}
\mathbb{R}^2 \\
\mathbb{R}^3
\end{array}$$

$$\begin{array}{c}
\mathbb{R}^1 \\
\mathbb{R}^1 \\
\mathbb{R}^1$$

[0046] The intermediate product which has [the inside of a formula, and R1, R2, R3 and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with a mild acid, and it is a formula and [0047].

[Formula 45]

[0048] The intermediate product which has [the inside of a formula, and R1 and B are as having given the definition previously] is formed, and it is iv. It is said intermediate product A formula and [0049] [Formula 46]

[0050] It is made to react under existence of the 2nd acid chloride and base which ****, and is a formula and [0051].

$$\begin{array}{c|cccc}
O & R^1 \\
\parallel & & \parallel \\
C - N - N - C - B \\
\parallel & & \parallel \\
H & O & (I)
\end{array}$$

[0052] The manufacture approach of an N and N'- diacyl-N'-t-alkyl hydrazine compound expressed with [the inside of a formula, and R1, A and B are as having given the definition previously] is offered. It sets in the more desirable mode of this invention, and is a structure expression [0053]. [Formula 48]

$$\begin{array}{c|cccc}
O & C(CH_3)_3 \\
I & I \\
I & I \\
H & O & (II)
\end{array}$$

[0054] Independently A and B among [type, respectively Phenyl or a halo, alkyl (C1-C2), (C1-C2) When [which was chosen from the group which consists of alkoxy ** and (C1-C2) alkoxy (C1-C2) alkyl] it is the same or the phenyl permuted by different 1 thru/or three different substituents and two adjoining locations of; phenyl ring are permuted by the alkoxy group] which these radicals become together and can form the dioxo llano or JIOKISANO heterocycle of 5 or 6 members, It is the manufacture approach of an N and N'- diacyl-N'-t-butyl hydrazine compound come out of and expressed, and is i. The acid addition salt to which t-butyl hydrazine or t-butyl hydrazine is equivalent, and formula [0055]

[0056] The ketone which **** is made to react under existence of a base, and it is a formula and [0057]. [Formula 50]

$$R^2$$
 $C = N - N - H$

[0058] The intermediate product which has [R2 is alkyl (C1-C2) among a formula, and R3 is alkyl (C1-C2)] is formed, and it is ii. It is said intermediate product in Process i A formula and [0059] [Formula 51]

[0060] It is made to react under existence of the acid chloride which ****, and a base, and is a formula and [0061].

[Formula 52]
$$\begin{array}{c}
C(CH_3)_3 \\
C = N - N - C - E
\end{array}$$

[0062] The intermediate product which has [the inside of a formula, and R2, R3 and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with the mild acid chosen from the group which consists of a maleic acid, an OKISA rucksack acid, phosphoric acid, an acetic acid, Para toluenesulfonic acid, formic acid, a chloroacetic acid, an alt.-nitro benzoic acid, a tartaric acid, and citric acid, and it is a formula and [0063].

[0064] The intermediate product which has [the inside of a formula and B are as having given the definition previously] is formed, and it is iv. It is said intermediate product A formula and [0065] [Formula 54]

[0066] It is made to react under existence of the 2nd acid chloride and base which ****, and is a formula and [0067].

(II)

[0068] The manufacture approach of an N and N'- diacyl-N'-t-butyl hydrazine compound of manufacturing the N and N'- diacyl-N'-t-butyl hydrazine compound expressed with [the inside of a formula, and A and B are as having given the definition previously] is offered. It sets in the most desirable mode of this invention, and is a structure expression and [0069]. [Formula 56]

[0070] A is phenyl, 2-fluoro phenyl, 4-chlorophenyl, 2-methyl-3-methoxypheny, or 4-ethyl phenyl among [type. B Phenyl, 3, 5-dimethylphenyl, 3-methylphenyl, 3-chlorophenyl, Or it is the manufacture approach of an N and N'- diacyl-N'-t-butyl hydrazine compound expressed with] which is 3 and 5-dichlorophenyl, and is i. The acid addition salt to which t-butyl hydrazine or t-butyl hydrazine is equivalent, An acetone is made to react under existence of a base and it is a formula and [0071]. [Formula 57]

[0072] The intermediate product which **** is formed and it is ii. It is said intermediate product in Process i A formula and [0073]

[Formula 58]

[0074] It is made to react under existence of the acid chloride which ****, and a base, and is a formula and [0075].

[Formula 59]

$$CH_3$$
 $C=N-N-C-B$
 CH_3
 CH_3

[0076] The intermediate product which has [the inside of a formula and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with the mild acid chosen from the group which consists of a maleic acid, an OKISA rucksack acid, phosphoric acid, an acetic acid, and Para toluenesulfonic acid, and it is a formula and [0077]. [Formula 60]

[0078] The intermediate product which has [the inside of a formula and B are as having given the definition previously] is formed, and it is iv. It is said intermediate product A formula and [0079] [Formula 61]

[0080] It is made to react under existence of the 2nd acid chloride and base which ****, and is a formula and [0081].

[0082] The manufacture approach of an N and N'- diacyl-N'-t-butyl hydrazine compound of manufacturing the N and N-diacyl-N-t-butyl hydrazine compound expressed with [the inside of a formula, and A and B are as having given the definition previously] is offered. The following examples are not shown for explanation of this invention, and do not restrict the range of this invention at all. [0083] A preparation process [of a -t-butyl-N-(4-ethyl benzoyl)-N'-(3, 5-dimethyl bay ZOIRU) hydrazine] i, and 1'N'-t-butyl hydrazone, [of iiN-isopropylidene-examples N] and N-isopropylidene-N -- '-t-butyl-N' -- formation of -3 and 5-dimethylbenzo ylhydrazone -- by the well-known approach An N-isopropylidene-N'-t-butyl hydrazone is formed from an acetone and t-butyl hydrazine hydrochloride. It is made to react with 3 and 5-dimethylbenzoyl chloride succeedingly, and is N-isopropylidene-N'-t-butyl. - N' - 3 and 5-dimethylbenzo ylhydrazone was formed. Process iiiN -- '-t-butyl-N' -3, the formation pars-basilaris-ossis-occipitalis draining valve of 5-dimethylbenzoyl hydrazine, an overhead agitator, a heating mantle, and addition -- 500ml 3 opening round bottom flask furnished with a funnel, and a condenser / Dean Stark equipment --N-isopropylidene-N -- '-t-butyl-N' -3, 5-dimethylbenzo ylhydrazone (99% of purity, 152 millimol), a maleic acid (17 millimol) and toluene, and 100ml of each water were thrown in. Mixture was heated, it was made to flow back and distilland was brought together in Dean Stark equipment. equipment is emptied periodically -- having -- addition -- a new solvent was added from the funnel. Mixture was distilled for a total of 197ml water, and 131ml toluene 5 hours after. When product mixture was analyzed by the gas chromatograph, it is mainly N'-t-butyl. - N' - It turned out that it is 3 and 5-dimethylbenzoyl hydrazine (94.6%). Other little components are N-isopropylidene-N'-t-butyl of starting material. - It is N'. - It was 3 and 5-dimethylbenzo ylhydrazone (3.2%). Process ivN'-t-butyl-N-4-ethyl benzoyl-N'-3, preparation N'-t-butyl-N'-3 of 5-dimethylbenzoyl hydrazine, 5-dimethylbenzoyl hydrazine, and 4-ethyl benzoyl chloride are made to react with a well-known means, and it is N'-t-butyl-N-4-ethyl benzoyl. - N' - 3 and 5-dimethylbenzoyl hydrazine was prepared. N-isopropylidene-N'-t-butyl [- The yield as the whole 3 and 5-dimethylbenzoyl hydrazine was 88%.] - N' - N'-t-butyl-N-4-ethyl benzoyl from 3 and 5-dimethylbenzo ylhydrazone - N' [0084] By the same approach as two to example 5 example 1, the shown aromatic series acid chloride was used and the following compounds were prepared. [0085]

An example An acid chloride A B 2 Benzoyl Benzoyl 3 2-fluoro benzoyl Benzoyl 4 4-chloro benzoyl Benzoyl 5 2-methyl-3- 3, 5-dimethyl - Methoxy benzoyl A benzoyl example Product 2 N-t-butyl-N and N'-dibenzoyl hydrazine 3 N-t-butyl-N-(2-fluoro benzoyl)- N'-benzoyl hydrazine 4 N-t-butyl-N-(4-chloro benzoyl)- N'-benzoyl hydrazine 5 N-t-butyl-N-(2-methyl-3-methoxy benzoyl)- N'-(3, 5-dimethylbenzoyl) hydrazine

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* NOTICES *

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- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.*** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

CLAIMS

R1 among [type the 3rd class (C4-C8) alkyl group, and A and B; halo which is independently chosen from phenyl, naphthyl, or the group that consists of the following radicals, respectively and which is the same, the phenyl permuted by different 1 thru/or three different substituents, or naphthyl, Cyano ** nitroglycerine, hydroxy ** mercapto, thiocyanate, alkyl (C1-C4), Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Amino, alkylamino (C1-C4), the JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently, Alkyl carbamoyl, the JI (C1-C4) alkyl carbamoyl in which each alkyl group has one to four carbon atoms independently, (C1-C4) Cyano (C1-C4) alkyl, alkoxy (C1-C4) (C1-C4) alkyl, The alkenyl, alkadienyl (C4-C6), alkynyl (C2-C6), (C2-C6) Alkyl dithio NETO, alkyl (C1-C4) carbonyl thio, (C1-C4) The Tori (C1-C4) alkyl silyl to which each alkyl group has one to four carbon atoms independently in a list, Phenyl and a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) In the phenyl which is chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which was permuted by the same or different 1 thru/or two substituents, phenoxy, and a list, a halo, Cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, alkoxy (C1-C4) ** Halo (C1-C2) alkyl, halo (C1-C2) alkoxy ** (C1-C4) alkylthio, Alkyl sulfinyl, an alkyl (C1-C4) sulfonyl, (C1-C4) Carboxy, the formyl, alkyl (C1-C4) carbonyl, alkoxy (C1-C4) carbonyl, Alkanoloxy, amino, alkylamino (C1-C4), (C1-C4) And the phenoxy which is chosen from the group which each alkyl group becomes from the JI (C1-C4) alkylamino which has one to four carbon atoms independently and which was permuted by the same or different 1 thru/or two substituents, In benzyl and a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Benzyl, phenoxy carbonyl which are chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which were permuted by the same or different 1 thru/or two substituents, In a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the

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formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Phenoxy carbonyl, phenylthio which are chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which were permuted by the same or different 1 thru/or two substituents, In a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) The phenylthio which is chosen from the group which consists of amino, alkylamino (C1-C4), and II (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and which was permuted by the same or different 1 thru/or two substituents, In phenyl (C1-C4) alkyl and a list, a halo, cyano ** nitroglycerine, hydroxy ** Alkyl, alkoxy (C1-C4) ** halo (C1-C2) alkyl, (C1-C4) Halo (C1-C2) alkoxy ** (C1-C4) alkylthio, alkyl (C1-C4) sulfinyl, An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Phenyl which is chosen from the group which consists of amino, alkylamino (C1-C4), and JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently and by which the phenyl ring was permuted by the same or different 1 thru/or two substituents (C1-C4) Alkyl; when two adjoining locations of a phenyl ring are permuted by the alkoxy group which these radicals become together and can form the dioxo llano (methylene dioxy) or JIOKISANO (1, 2-ethylene dioxy) heterocycle of 5 or 6 members, An N [which is come out of and expressed], and N'- diacyl-N'-t-alkyl hydrazine compound, Or it is the manufacture approach of the salt permitted agriculturally, and is i. The acid addition salt to which a formula, the 3rd class (C4-C8) alkyl hydrazine expressed with H2NNHR1, or this the 3rd class (C4-C8) alkyl hydrazine is equivalent, and formula [** 2]

The ketone or aldehyde which **** is made to react under existence of a base, and they are a formula and [Formula 3].

$$C = N - N - H$$

The intermediate product which has [R1 is as having given the definition previously among a formula, R2 is a hydrogen atom or (C1-C3) alkyl, and R3 is alkyl (C1-C6)] is formed, and it is ii. About said intermediate product in Process i, they are a formula and [Formula 4].

It is made to react under existence of the acid chloride which ****, and a base, and they are a formula and [Formula 5].

$$R^{2} C = N - N - C - E$$

$$R^{3} C = N - N - C - E$$

The intermediate product which has [the inside of a formula, and R1, R2, R3 and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with a mild acid, and they are a formula and [Formula 6].

The intermediate product which has [the inside of a formula, and R1 and B are as having given the definition previously] is formed, and it is iv. About said intermediate product, they are a formula and [Formula 7].

It is made to react under existence of the 2nd acid chloride and base which ****, and they are a formula and [Formula 8].

It is the aforementioned manufacture approach which forms an N [which is expressed with [the inside of a formula, and R1, A and B are as having given the definition previously]], and N'- diacyl-N'-t-alkyl hydrazine compound.

[Claim 2] R1 is t-butyl, 1, and 1-dimethyl pentyl or 1,1,3,3-tetrametylbutyl. A and B; halo which is independently chosen from phenyl, naphthyl, or the group that consists of the following radicals, respectively and which is the same, the phenyl permuted by different 1 thru/or three different substituents, or a naphthyl group, Cyano ** nitroglycerine, thiocyanate, alkyl (C1-C4), alkoxy (C1-C4) ** Halo (C1-C2) alkyl, halo (C1-C2) alkoxy ** (C1-C4) alkylthio, Alkyl sulfinyl, an alkyl (C1-C4) sulfonyl, (C1-C4) Carboxy, the formyl, alkyl (C1-C4) carbonyl, alkoxy (C1-C4) carbonyl, Alkanoloxy, the JI (C1-C4) alkylamino in which each alkyl group has one to four carbon atoms independently, (C1-C4) Carbamoyl, alkyl (C1-C4) carbamoyl, the JI (C1-C4) alkyl carbamoyl in which each alkyl group has one to four carbon atoms independently, Cyano (C1-C4) alkyl, alkoxy (C1-C4) (C1-C4) alkyl, The alkenyl, alkadienyl (C4-C6), alkynyl (C2-C6), (C2-C6) Alkyl dithio NETO, alkyl (C1-C4) carbonyl thio, (C1-C4) The Tori (C1-C4) alkyl silyl in which each alkyl group has one to four carbon atoms independently, Phenyl, phenoxy, bay ZOIRU, phenyl (C1-C2) alkyl, In a list, a halo, cyano ** nitroglycerine, hydroxy ** (C1-C4) alkyl, Alkoxy ** halo (C1-C2) alkyl and halo (C1-C2) alkoxy ** (C1-C4) Alkylthio, alkyl (C1-C4) sulfinyl, (C1-C4) An alkyl sulfonyl, carboxy, the formyl, alkyl (C1-C4) carbonyl, (C1-C4) Alkoxy carbonyl, alkanoloxy (C1-C4), (C1-C4) Each alkyl group independently and one to four carbon atoms Phenyl (C1-C2) alkyl which is chosen from the group which consists of JI (C1-C4) alkylamino which it has and by which the phenyl ring was permuted by the same or different 1 thru/or two substituents; when two adjoining locations of a phenyl ring are permuted by the alkoxy group I which these radicals become together and can form the dioxo llano or JIOKISANO heterocycle of 5 or 6 members, An N [which is come out of and expressed], and N'diacyl-N'-t-alkyl hydrazine compound, Or it is the manufacture approach of the salt permitted agriculturally, and is i. The acid addition salt to which a formula, the 3rd class (C4-Ĉ8) alkyl hydrazine expressed with H2NNHR1, or this the 3rd class (C4-C8) alkyl hydrazine is equivalent, and formula [** 91

The ketone which **** is made to react under existence of a base, and they are a formula and [Formula 10].

$$R^{2} C = N - N - H$$

The intermediate product which has [R1 is as having given the definition previously among a formula, R2 is alkyl (C1-C3), and R3 is alkyl (C1-C4)] is formed, and it is ii. About said intermediate product in

Process i, they are a formula and [Formula 11].

It is made to react under existence of the acid chloride which ****, and a base, and they are a formula and [Formula 12].

The intermediate product which has [the inside of a formula, and R1, R2, R3 and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with a mild acid, and they are a formula and [Formula 13].

The intermediate product which has [the inside of a formula, and R1 and B are as having given the definition previously] is formed, and it is iv. About said intermediate product, they are a formula and [Formula 14].

It is made to react under existence of the 2nd acid chloride and base which ****, and they are a formula and [Formula 15].

It is the manufacture approach of an according to claim 1 N [which forms the N and N'-diacyl-N'-t-alkyl hydrazine compound expressed with [the inside of a formula, and R1, A and B are as having given the definition previously]], and N'-diacyl-N'-t-alkyl hydrazine compound. [Claim 3] Structure expression [** 16]

Independently A and B among [type, respectively Phenyl or a halo, alkyl (C1-C2), (C1-C2) When [which was chosen from the group which consists of alkoxy ** and (C1-C2) alkoxy (C1-C2) alkyl] it is the same or the phenyl permuted by different 1 thru/or three different substituents and two adjoining locations of; phenyl ring are permuted by the alkoxy group] which these radicals become together and can form the dioxo llano or JIOKISANO heterocycle of 5 or 6 members, It is the manufacture approach of an N and N'- diacyl-N'-t-butyl hydrazine compound come out of and expressed, and is i. The acid addition salt to which t-butyl hydrazine or t-butyl hydrazine is equivalent, and formula [** 17]

The ketone which **** is made to react under existence of a base, and they are a formula and [Formula 18].

The intermediate product which has [R2 is alkyl (C1-C2) among a formula, and R3 is alkyl (C1-C2)] is formed, and it is ii. About said intermediate product in Process i, they are a formula and [Formula 19].

It is made to react under existence of the acid chloride which ****, and a base, and they are a formula and [Formula 20].

$$R^2$$
 $C = N - N - C - B$
 R^3
 $C = N - N - C - B$

The intermediate product which has [the inside of a formula, and R2, R3 and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with the mild acid chosen from the group which consists of a maleic acid, an OKISA rucksack acid, phosphoric acid, an acetic acid, Para toluenesulfonic acid, formic acid, a chloroacetic acid, an alt.-nitro benzoic acid, a tartaric acid, and citric acid, and they are a formula and [Formula 21].

The intermediate product which has [the inside of a formula and B are as having given the definition previously] is formed, and it is iv. About said intermediate product, they are a formula and [Formula 22].

It is made to react under existence of the 2nd acid chloride and base which ****, and they are a formula and [Formula 23].

It is the manufacture approach of an according to claim 2 N [which manufactures the N and N'-diacyl-N'-t-butyl hydrazine compound expressed with [the inside of a formula, and A and B are as having given the definition previously]], and N'- diacyl-N'-t-alkyl hydrazine compound. [Claim 4] A is phenyl, 2-fluoro phenyl, 4-chlorophenyl, 2-methyl-3-methoxypheny, or 4-ethyl phenyl, B is phenyl, 3, 5-dimethylphenyl, 3-methylphenyl, 3-chlorophenyl or 3, and 5-dichlorophenyl, and it is i. An acetone is made to react under existence of a base and they are [the acid addition salt to which t-butyl hydrazine or t-butyl hydrazine is equivalent, and] a formula and [Formula 24].

$$CH_3$$
 $C=N-N-H$ CH_3

The intermediate product which **** is formed and it is ii. About said intermediate product in Process i, they are a formula and [Formula 25].

It is made to react under existence of the acid chloride which ****, and a base, and they are a formula and [Formula 26].

$$CH_3$$
 $C=N-N-C-B$
 CH_3
 CH_3

The intermediate product which has [the inside of a formula and B are as having given the definition previously] is formed, and it is iii. Said intermediate product in Process ii is made to react with the mild acid chosen from the group which consists of a maleic acid, an OKISA rucksack acid, phosphoric acid, an acetic acid, and Para toluenesulfonic acid, and they are a formula and [Formula 27].

The intermediate product which has [the inside of a formula and B are as having given the definition previously] is formed, and it is iv. About said intermediate product, they are a formula and [Formula 28].

It is made to react under existence of the 2nd acid chloride and base which ****, and they are a formula and [Formula 29].

It is the manufacture approach of an according to claim 3 N [which manufactures the N and N'-diacyl-N'-t-butyl hydrazine compound expressed with [the inside of a formula, and A and B are as having given the definition previously]], and N'- diacyl-N'-t-butyl hydrazine compound. [Claim 5] N'-t-butyl-N, the N'-dibenzoyl hydrazine which were manufactured by the approach according to claim 1, An N'-t-butyl-N-(2-fluoro benzoyl)-N'-benzoyl hydrazine, An N'-t-butyl-N-(2-methyl-3-methoxy benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, An N'-t-butyl-N-(4-ethyl benzoyl)-N'-(3, 5-dimethylbenzoyl) hydrazine, And the N and N'-diacyl-N'-t-alkyl hydrazine compound chosen from the group which consists of an N'-t-butyl-N-(4-chloro benzoyl)-N'-benzoyl hydrazine.

[Translation done.]